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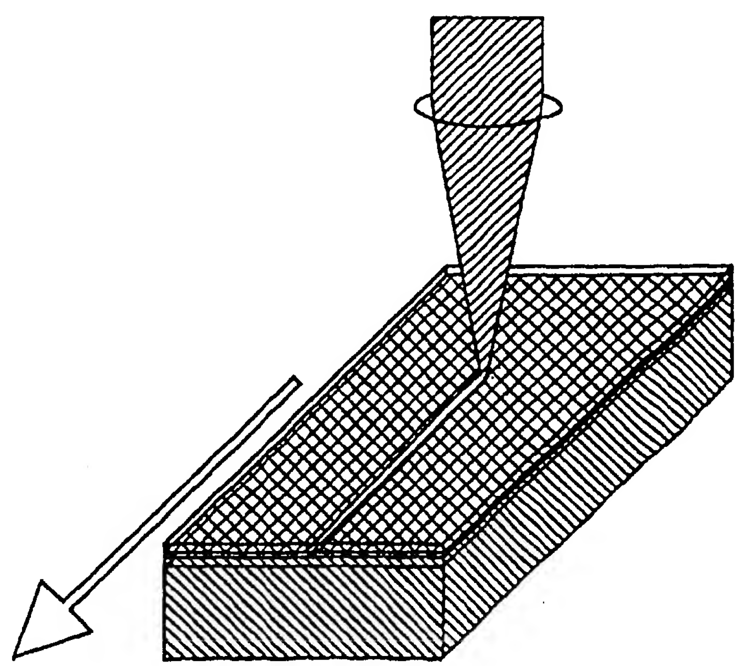
(54) Title: A METHOD OF MANUFACTURING GERMANIUM DOPED GLASSES AND A USE OF THE METHOD

(57) Abstract

A method of manufacturing germanium doped glasses, the refractive index of which can be changed preferably by exposure to UV-light by depositing a gas mixture. According to the invention, compounds containing N and H, such as NH_3, which may be incorporated into the glass, are added to the gas mixture to be deposited. The invention further relates to the use of the method for the manufacture of an optical transducer, utilizing the fact that the filter frequency or frequencies is/are changed in response to the strain of the glass material.



Title: A method of manufacturing germanium doped glasses and a use of the method.

Technical Field

The invention relates to a method of manufacturing germanium doped glasses, the refractive index of which may be changed preferably by exposure to UV-light by depositing a gas mixture.

Background Art

It is known that the refractive index of germanium doped glasses may be changed by means of ultra-violet light, confer WO95/26519. The changes in question are of 10^{-5} - 10^{-2}. The higher the content of germanium, the larger the changes. At a content of germanium of about 20% or more the properties of the glass deteriorate.

It is further known to add H_2 to the glass to improve the photosensitivity, confer EP 569182 and JP-07244210.

Brief Description of the Invention

The object of the invention is to show how the photosensitivity of the glass may be further improved without thereby deteriorating the other properties of the glass.

According to the invention, this object is obtained by adding compounds of N and H, such as NH_3 which may be incorporated into the glass. Hydrogen and/or nitrogen can thus compensate for the quality deterioration possibly caused by the germanium doping. At the same time, glasses are obtained, in which in particular by means of UV-light larger refractive index changes can be induced than hitherto known.

Moreover, according to the invention, F and/or B can be added to the gas mixture

to be deposited, whereby an enhanced control of the refractive index is possible.

The method may for instance be used for UV-writing of active waveguides by adding rare earth ions, such as Er, Yb, Nd or Pr to the gas mixture to be deposited for UV-writing of the active waveguides.

The invention further relates to a use of the method of manufacturing an optical transducer. When focusing two coherent beams down to such a waveguide, an interference pattern of index changes is provided, whereby the particular light guide can be used as a filter. This filter can in turn be used as a strain, temperature and pressure transducer, utilizing the fact that the filter frequency or frequencies change(s) in response to the strain of the glass material. The transducer may for instance be part of an accelerometer.

Brief Description of the Drawings.

The invention is explained in greater detail below with reference to the accompanying drawings in which

Fig. 1 illustrates the mask method for the manufacture of optical waveguides,

Fig. 2 illustrates the method according to the invention by direct UV-writing for the manufacture of optical waveguides,

Fig. 3 shows a set-up to be used in connection with the writing method illustrated in Fig. 2,

Fig. 4a and 4b are illustrations of the intensity distribution of Y-splitters made by the writing method according to the invention,

Fig. 5 shows an accelerometer made by the method according to the invention,

Fig. 6 illustrates a reactor for the deposition of the glass layers to be written in, and

Fig. 7 illustrates an impedance matching unit of the reactor.

Best Mode for Carrying Out the Invention

It has been found that it is possible to provide a permanent change in the refractive index of germanium doped glasses by means of ultra-violet light, in particular. Moreover, it has been found that photo-induced gratings may be directly written in germanium silicate optical fibres when focusing two coherent interfering UV-laser beams.

Furthermore, the photosensitivity of germanium doped glasses depends widely on the germanium concentration, confer R. J. Campbell and Kashyab "The properties and applications of photosensitive germanosilicate fibre" International Journal of Optoelectronics 1994, vol 9, No. 1, pp. 33-57. The fact that germanium doped glasses are partially soluble in water when having a high germanium content (in excess of about 20%), limits the effect obtained by increasing the Ge-concentration. Index changes of up to 3×10^{-3} have been found for Ge-doped PECVD-glasses, confer Christian V. Poulsen, Jörg Hübner, Karin Zenth et al. "UV-writing of Integrated Optical Components".Moreover, hydrogen loading of Ge-doped glass has been found to enhance the photosensitivity. The long-term stability is, however, questionable, and the OH-induced transmission losses in the infrared area are increased significantly. The optimum index change obtainable by this method is 5×10^{-3}.

Finally, co-doping with boron has proved to increase the photosensitivity of Ge-doped optical fibres. Index changes of up to 1.5×10^{-3} have been obtained by this method. However, the combination of hydrogen loading and boron co-doping has resulted in index changes of up to 7.5×10^{-3}, confer G.D. Maxwell, B.J. Ainslie. "Demonstration of a directly written directional coupler using UV-induced photosensitivity in a planar silica waveguide", Electronic Letters 19 Jan. 1995, vol 31, No.

2 pp. 95- 96. The glass quality is not mentioned.

Advanced optical networks based on planar waveguides are expected to be general components in future telecommunication networks. Optical sensor systems are also expected to be based on the planar waveguide technology.

Typically, the known methods of producing planar waveguides comprise complicated and time-consuming processing steps, such as photolithography, reactive ion etching and reflowing of glass. Direct laser writing of planar waveguides in photosensitive glasses may simplify the process significantly, and consequently, a more flexible fabrication in terms of the design of planar waveguide networks is possible. A film waveguide formed of a three-layer glass structure on silicon may be made by means of Plasma Enhanced Chemical Vapour Deposition (PECVD). The glass layer structure is formed of a buffer, a core and a cladding, the core material being photosensitive, cf. G.D. Maxwell and B.J. Ainslie, "Demonstration of a directly written directional coupler using UV-induced photosensitivity in a planar silica waveguide", Electronics Letters 19 January 1995, vol 31, No 2, pp. 95-96.

In order to minimize the coupling loss when coupling planar waveguides to optical fibres, the mode profiles of the two waveguides have to be the same. A difference of 5×10^{-3} in the refractive indices of the cladding and the core of the planar waveguide is preferable to ensure low loss coupling to standard optical fibres.

By adding NH_3 or other gasses containing compounds of N and H to the gas mixture used for PECVD of Ge-doped glasses, UV-induced index changes exceeding 3.5×10^{-3} have been obtained without hydrogen loading. This co-doping with nitrogen in form of NH_3 also improves the quality of the glass. The particles in the glass are thus presumably smaller, which may result in reduced scattering losses in for instance optical waveguides. Although, typical Ge-doped glasses may have a refractive index ranging from 1.46 to 1.485 without being water-soluble, this glass ranges from 1.46 to 1.51 or above. A co-doping with nitrogen reduces the stresses in the glass. Stress-free glasses deposited at 300°C have been developed. In the

optical waveguides it is preferable to minimize stress-induced birefraction, in particular in optomechanical micro devices, such as bridge structures, confer Danish utility model No. 9500197.

A method of the manufacture of a two-dimensional film waveguide is described in the following. A two-dimensional film waveguide comprising a three-layer structure (buffer, core and cladding) on a silicon wafer may be made by Plasma Enhanced Chemical Vapour Deposition (PECVD), confer Fig. 1. According to the known methods the manufacture of buried channel waveguides and more advanced optical components involves time-consuming processing step such as photolithography, reactive ion etching and reflowing of the glass. These processing steps may be avoided by employing a UV-writing technique provided a fast and simple production method for prototypes and small quantities. However, this technique can also be used for mass production.

Two methods of providing integrated optical components are compared in the following. The methods are dependent on the UV-sources used. By means of a stable CW-source having a mode profile corresponding to TEM_{00}, a waveguide can be induced by focusing the UV-beam down to the desired width of the waveguide and drawing the desired structure by moving the film waveguide under the beam by means of a highly accurate XY-translation stage. This method is denoted as direct UV-drawing of waveguides, confer Fig. 2. When using a powerful excimer source, a mask having the desired structure is to be used, confer Fig. 1. The advantages and drawbacks of each said method are discussed below.

<u>Deposition technique</u>

The glass was deposited in a mixture of SiH_4, GeH_4, NH_3, N2 and N_2O at 300°C in a special reactor, confer Fig. 6. Due to the gas mixture the hydrogen was incorporated into the deposited glass. Subsequently, hydrogen was partly removed by annealing the glass at 800°C. Compared to a flame hydrolysis, in which the glass temperature reaches approximately 2000°C, the PECVD is a low temperature

process providing a better control of the glass matrix. Major UV-induced index changes without hydrogen loading have been observed. Three layers of silicon dioxide were deposited with a thickness of 12 μm, 2.5 μm and 12 μm, respectively. The core layer thickness of 2.5 μm served to obtain a single mode operation in vertical direction, and the cladding and buffer layer thickness of 12 μm served to prevent a coupling to the surroundings. The GeO_2 concentration in the core layer was 14 mole%. The refractive index change saturates at about 3.5 x 10^{-3} without hydrogen loading. However, such refractive index changes are sufficient to produce most of the standard components.

A frequency doubled Ar-ion-CW-laser was used in the UV-drawing set-up shown in Fig. 3. The beam profile corresponded essentially to the TEM_{00} mode, which made it possible to focus a beam down to a width of 5 μm with only one focusing lens. The beam may be focused down to 1 μm by means of diffraction limited optics. The laser power was about 100 mW providing a maximum intensity of approximately 500 kW/cm^2 in a beam having a diameter of 5 μm.

The design of the waveguide is obtained by means of a CAD program and transferred to the film waveguide via a computer controlled XY-stage. The XY-stage used was a Newport PM 500 system with a precision of 100 nm over 10 x 10 cm^2, whereby waveguides may be induced across an entire 4" wafer with high accuracy. As a result, a considerably more flexible tool is obtained than hitherto known, the time interval from idea to prototype being very short. The width of the UV-beam and consequently, the width of the induced waveguides, is controlled by defocusing the UV-beam by means of a Z-stage supporting the focusing lens. By changing the focus, the intensity of the UV-light on the wafer may be changed. If changes in dimensions are needed, possibly a narrowing or widening thereof, either the intensity or the writing rate of the UV-light is to be controlled or the refractive index change is to be saturated.

In the second set-up, confer Fig. 1, a pulsed excimer laser is used yielding light at a wavelength of 193 nm. The pulse energy is about 100 mJ at a pulse rate of 20 ns.

The beam is a multi-mode beam, typically comprising 10^5 to 10^6 modes and various divergences in the horizontal and vertical planes. At the output of the laser, the spot size of the beam is approximately 25 x 8 mm^2. A vibrating quartz plate was inserted into the beam to average the mode profile. The spot size in vertical direction was reduced by means of an anamorphic Galilei beam compressor. The spot size was reduced to about 36 x 2 mm^2 resulting in an intensity of about 80 mJ/cm^2. This spot size was used to expose the film waveguide, confer Fig. 3.

The vapour deposited film waveguide is covered by an aluminium layer, in which a pattern is provided by means of photolithography and wet etching. Due to its extremely high reflectivity at 193 nm, the use of aluminium as a mask material is particularly advantageous. In addition, the use of thin aluminium mask layers is a well-established technology.

Table 1

	Direct UV-writing	Mask method
Laser requirements	TEM_{00} UV-laser mode 244 nm FreD	Pulsed laser 193 nm, 248 nm
Advantages	Fast prototyping Flexible	Uncritical alignment Highly reproducible
Draw-backs	Need careful alignment Laser noise is transferred	Limited by mask design Additional wafer processing

In Table 1, a comparison between the two methods is shown. The laser requirements as well as the advantages and drawbacks of the methods are stated. Due to the direct UV-drawing, the design of the component is almost completely without restrictions and thus only limited by the maximum obtainable index value. The alignment is, however, critical and any fluctuations of the laser may effect the performance of the end component. The mask method is advantageous in that an uncritical alignment and exposure or scanning and any laser fluctuations are equalized ensuring that reproducible results may still be obtained.

In the set-up for direct UV-writing, straight waveguide initially indicated that the width of the waveguides was controllable and the drawings rates of about 20 - 50 μm/sec. were sufficient. However, the drawing time depends on the glass samples and said time may be reduced by hydrogen loading at the expense of a broad OH-resonance band at 1400 nm. The intensity was controlled to obtain so-called type I effects to reduce the transmission loss. A transmission loss of 0.2 dB/cm over a broad wavelength range was measured comparable with the transmission losses in conventionally produced planar waveguides. Subsequent to checking the drawing technology, more advanced components were drawn, such as couplers and Y-splitters. A Y-splitter with a 50:50 coupling ratio has been experienced, confer Fig. 4. The splitting ratio was, however, sensitive to the input alignment indicating the multi-mode waveguides. The directional couplers were also sensitive to input alignment. This indicates that the UV-induced change of the refractive index exceeded 6×10^{-3} with hydrogen loading. However, the excess loss was less than 0.2 dB, which indicates that the coupling areas were written with great accuracy.

Y-splitters written with the 193 nm excimer laser and an aluminium mask were evaluated on an automatic alignment set-up. In this set-up, semiconductor lasers are used operating at wavelengths of 1552 nm and 1310 nm, respectively. The splitting ratio was measured to be 50:50 at 1541 nm and to be 51:49 at a wavelength of 1310 nm. Fig. 4 illustrates the intensity distribution of the two splitter arms at 1542 nm and at 1310 nm, respectively.

The transmission loss is less than 2.8 dB/cm indicating type II index changes and deformation of the glass. Any differences in propagation loss between the Y-splitter and the straight waveguides UV-written under the same conditions were not measured. This indicates that there are only very small losses in a bending when a 5 mm radius of curvature is used to separate the splitter arms. The UV-induced change in the refractive index was estimated to be at least 3.5×10^{-3} without hydrogen loading after exposure with 4800 J/cm^2.

Table 2

	Direct UV-writing	Mask method
Intensity per area	5-500 kW/cm^2 (d: 5-50 μm)	5 MW/cm^2 (for all widths)
Energy per area	~ 1-100 kJ/cm^2 (d: 5-50 μm)	~ 0,3 kJ/cm^2
Time per length	200-500 s/cm (20-50 $\mu m/s$)	120 s/cm (80 $\mu m/s$)
Time per area	~ 10^6 s/cm^2 (~ 100$\mu m^2/s$)	600 s/cm^2 (~ 1,7·$10^6 \mu m^2/s$) (2)

A comparison between the two set-ups is shown in Table. 2. The index change is a non-linear function of the intensity of the UV-light. Due to the high intensity of the excimer laser, the mask method is, however, substantially faster, in particular when writing large areas.

The PECVD deposition system shown in Fig. 6 comprises a capacitively coupled reactor connected with an impedance matching unit. The impedance of the gas discharge is dependent on the discharge conditions, such as the discharge frequency, the type of gas, pressure and power density. In normal operation of the 380 kHz deposition system, this results in plasma series resistances in the range of 200 - 700 Ω and series capacitive reactance in the range of 5 - 2000 Ω. An impedance match is necessary to couple power from the generator with an impedance of 50 Ω to avoid reflections. The impedance matching unit is illustrated in Fig. 7.

The glass film is deposited on a substrate 3 arranged on an internal, capacitively coupled electrode 2 of the reactor connected to a 380 kHz generator.

The reactor is a parallel plate reactor, in which the chamber walls and an electrode in form of a substrate holder 12 are grounded. The reactor is impedance matched to minimize the reflections to the reactor. The impedance matching is carried out automatically by means of feedback loops.

The RF signal from the generator is fed to a top electrode 4 in the reactor through the impedance matching unit. The two electrodes 2,4 are preferably circular, about 25 cm in diameter and interspaced by 22.0 mm. The chamber walls and the two electrodes 2, 4 are made of aluminium. The top electrode 4 is insulated from the chamber walls.

The substrate holder 2 is kept at a temperature between 300 and 400°C, while the top electrode 4 is kept at a temperature of about 250°C.

The process gasses are fed into the reactor chamber through an aperture formed as a shower head in the top electrode 4. As a result, an even distribution of the gasses is ensured, whereby the deposition on the substrate 3 becomes very uniform. The gasses are fed into the reactor chamber through a stainless steel pipe 5 of a mass flow controller (not shown). A pump having a capacity of 1000 l/min is used to evacuate the reactor chamber. The base pressure of the chamber is approximately 0.1 mTorr. The purity of the process is due to the high gas flow rate (2000-3000 Sccm), whereby the purity is determined by the purity of the gasses and not by the base pressure of the chamber.

The deposition process in the chamber is controlled by means of a computer connected to the impedance matching unit. Consequently, a highly stable process is obtained (with a view to the pressure, the generator power, the impedance matching unit, the gas flow, the substrate temperature, the deposition time, etc.).

A standard process was run prior to the actual tests in order to monitor the conditions in the reactor chamber. The standard process is a 500 mTorr discharge of 17 Sccm SiH_4, 833 Sccm N_2, 1700 Sccm N_2O at a generator power of 500 W.

In a special embodiment the operating parameters are as follows:

	Ge-doped glass Type A (Low Power)	Process conditions
Flow (sccm)	SiH_4	15
	GeH_4	2
	$(SiH_4 + GeH_4)$	17
	N_2	833
	N_2O	1600
	NH_3	400
RF Power (W)	lf 380 kHz	380
Pressure (mTorr)		400
Temperature (°C)		300

Glass type:	Type A
GeH/ $(GeH_4 + SiH_4)$	≈ 12%
NH_3 flow (sccm)	400
RF Power (W)	380
Pressure (mTorr)	400
UV-induced Δn	$> 3.5 \times 10^{-3}$

By monitoring the refractive index, the deposition rate and the settings of the impedance matching unit, the reproducibility of the process can be checked.

The deposition may optionally be performed by flame hydrolysis. Other dopans, such as F or B, can be added to the gas mixture to the deposited.

The deposition can be substituted by an ion exchange or an ion implantation. Rare earth ions, such as Er, Yb, Nd or Pr can be added to the gas mixture to be deposited. As a result, active waveguides and non-linear components can be produced.

The method according to the invention can be used for the manufacture of diffractive elements, such as filters, gratings for coupling of light and reflectors and lenses.

The diffractive elements can be made by means of a holographic element, such as a phase mask or a prism. The method can also be used for the manufacture of interferometers and integrated optics in general.

There are an almost infinite number of usable compounds containing N and H, among others:

Inorganic:	Ammonium acetate $NH_4C_2H_3O_2$
	Ammonium cyanide NH_4CN

More than 180 different compounds containing ammonium are mentioned in the "CRC Handbook of Chemistry and Physics", 74th edition, pp. 4-37 to 4-40. The compounds are a mixture of gasses, liquids and solid substances depending on the pressure and temperature.

Organic substances:	Acenaphthene $C_{12}H_{11}N$
	Acetaldehyde $CH_3CN{=}NNHC_6H_5$

Further examples are rendered in said Handbook. Liquids are also relevant, as Solution Doping can be used or liquids can be fed through a nozzle with to atomization. Both said methods are used at the manufacture of thin glass films. Solid substances can also be used by heating the substance in connection with a passing carrier gas.

Claims

1. A method of manufacturing germanium doped glasses, the refractive index of which can be changed preferably by exposure to UV-light by depositing a gas mixture, c h a r a c t e r i s e d in that compounds containing N and H, such as NH_3, which may be incorporated into the glass, are added to the gas mixture to be deposited.

2. A method as claimed in claim 1, c h a r a c t e r i s e d in that F and/or B is added to the gas mixture to be deposited.

3. A method as claimed in claim 1, c h a r a c t e r i s e d in that rare earth ions, such as Er, Yb, Nd or Pr are added to the gas mixture to be deposited for UV-writing of for instance active waveguides.

4. The use of the method according to claim 1 for the manufacture of an optical transducer, utilizing the fact that the filter frequency or frequencies is/are changed in response to the strain of the glass material.

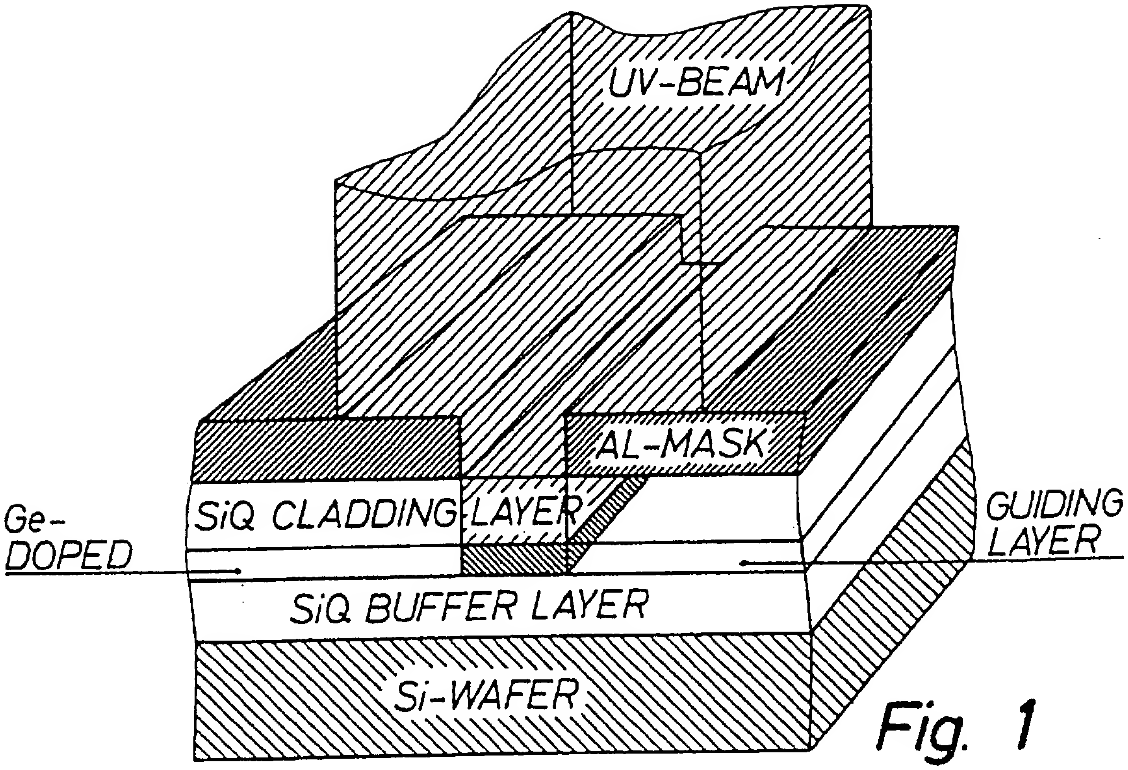

UV-BEAM
AL-MASK
SiQ CLADDING LAYER
Ge-
DOPED
GUIDING
LAYER
SiQ BUFFER LAYER
Si-WAFER

Fig. 1

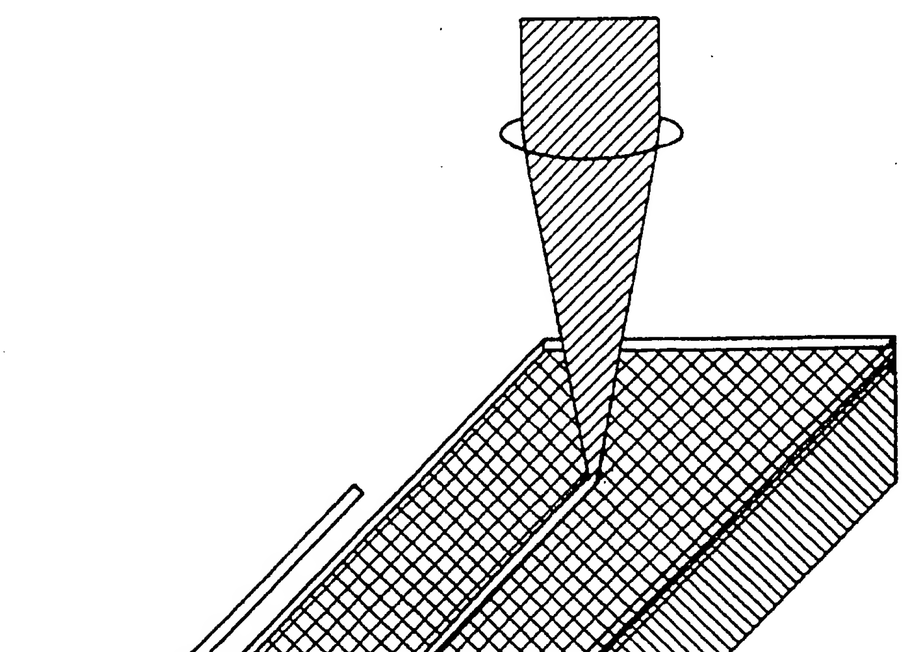


Fig. 2

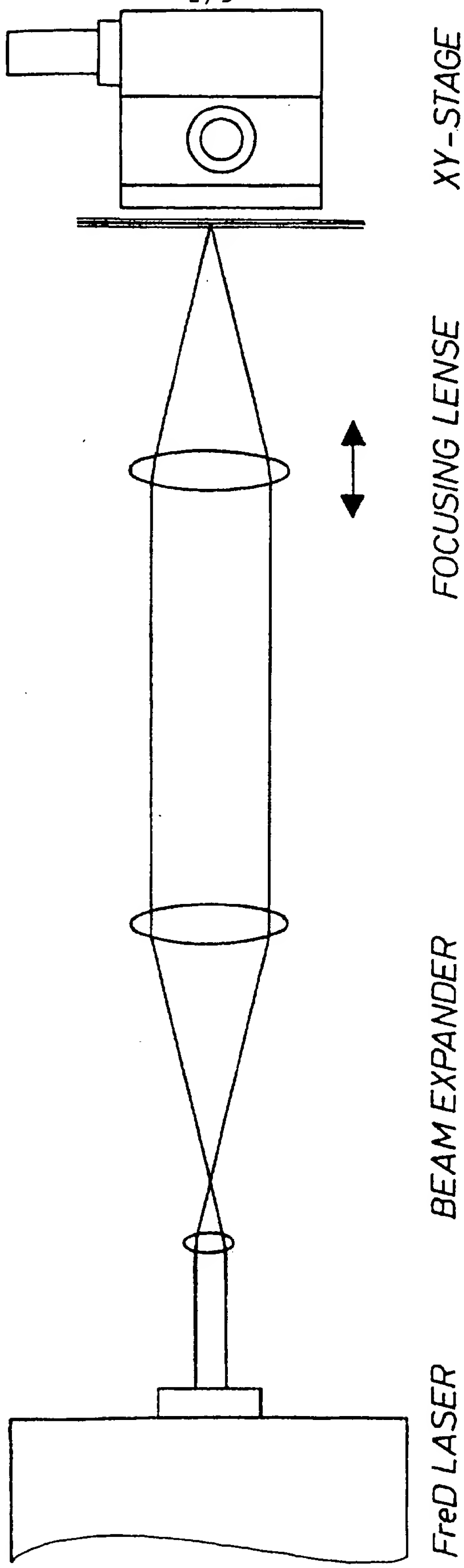


Fig. 3

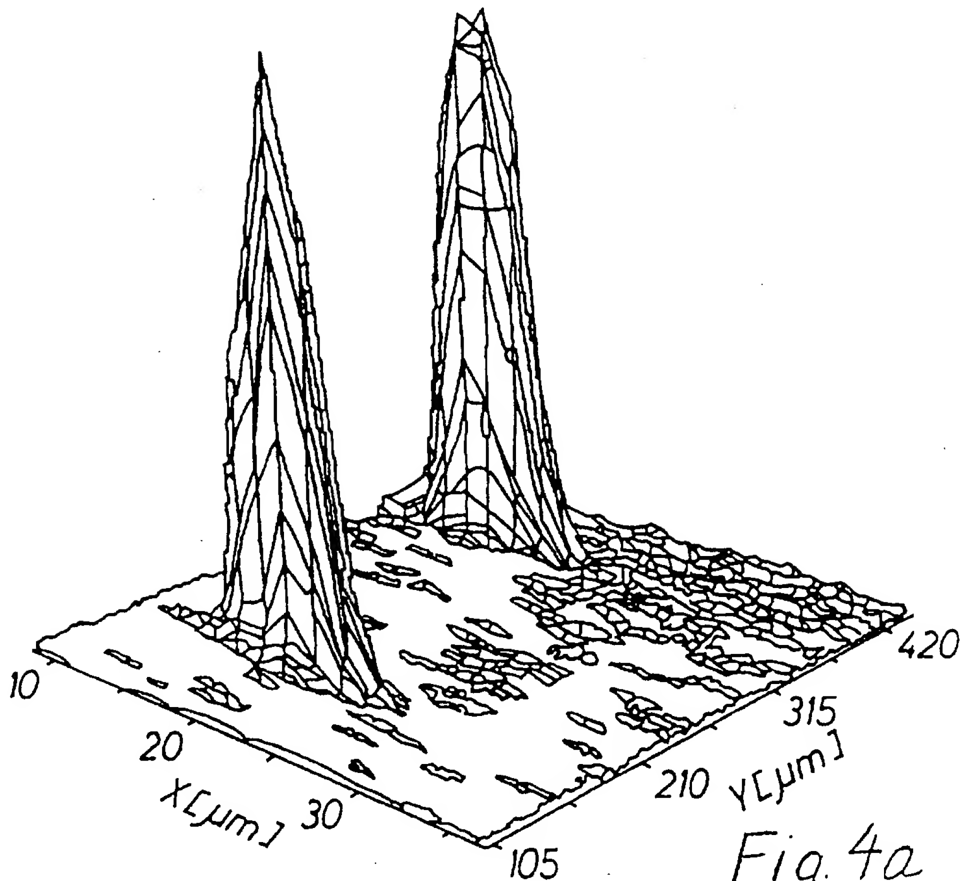


Fig. 4a

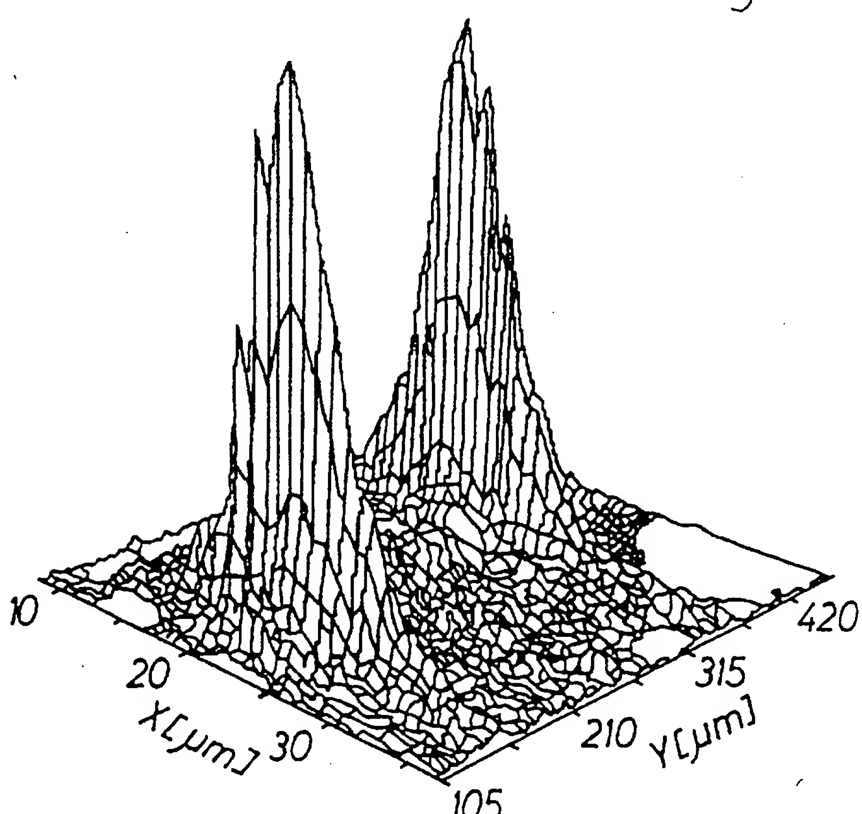


Fig. 4b

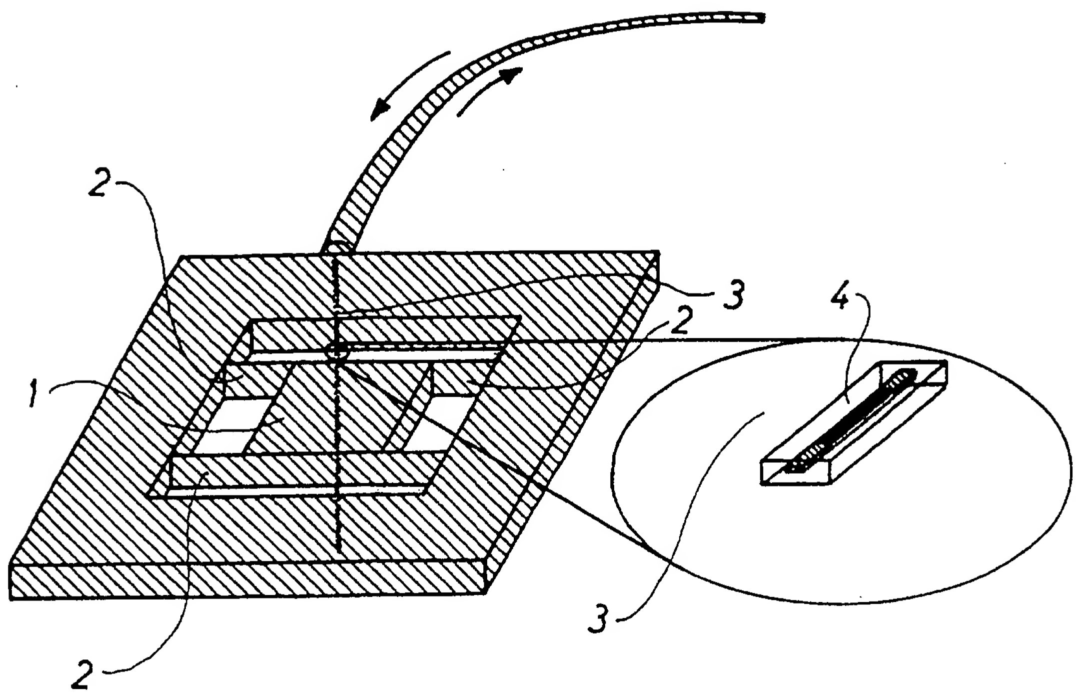


Fig. 5

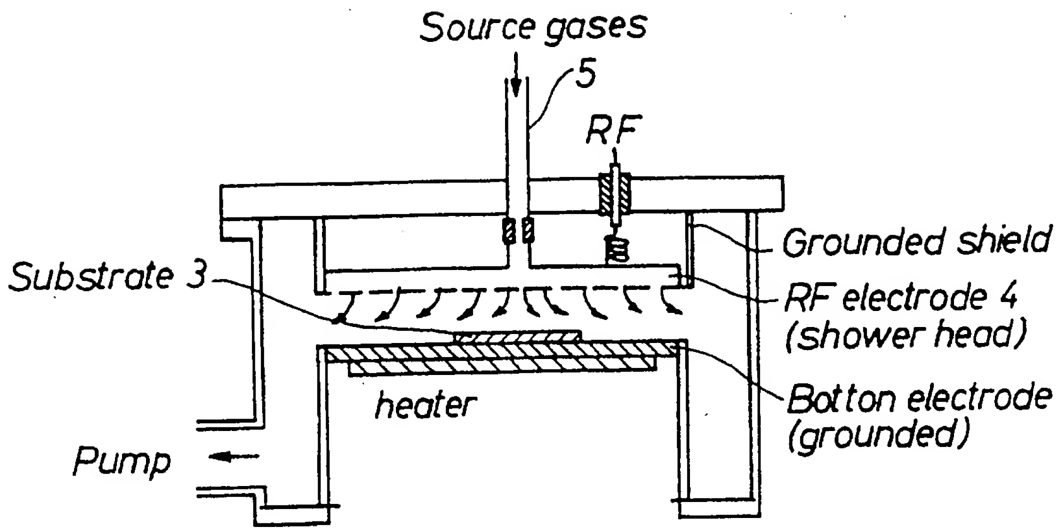

Source gases
5
RF
Grounded shield
Substrate 3
RF electrode 4
(shower head)
heater
Botton electrode
(grounded)
Pump

Fig. 6

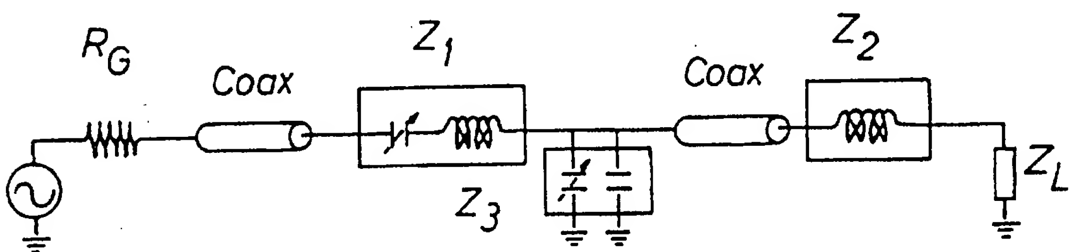

R_G
Coax
Z_1
Coax
Z_2
Z_3
Z_L

Fig. 7

INTERNATIONAL SEARCH REPORT

International application No.
PCT/DK 96/00517

A. CLASSIFICATION OF SUBJECT MATTER

IPC6: C03C 21/00, C03C 25/02 // G02B 6/16
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC6: C03C, G02B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

DIALOG: WPI, CLAIMS

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 0622343 A2 (AT & T CORP.), 2 November 1994 (02.11.94), abstract -- --------	1-4

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
14 March 1997	05 April 1997 (05.04.97)
Name and mailing address of the ISA/ Swedish Patent Office Box 5055, S-102 42 STOCKHOLM Facsimile No. +46 8 666 02 86	Authorized officer May Hallne Telephone No. +46 8 782 25 00

INTERNATIONAL SEARCH REPORT
Information on patent family members

03/02/97

International Application No.
PCT/DK 96/00517

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP-A2- 0622343	02/11/94	JP-A- 6345475	20/12/94
		US-A- 5478371	26/12/95
		US-A- 5500031	19/03/96

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